Reactor Utilization in Support of Research and Technology

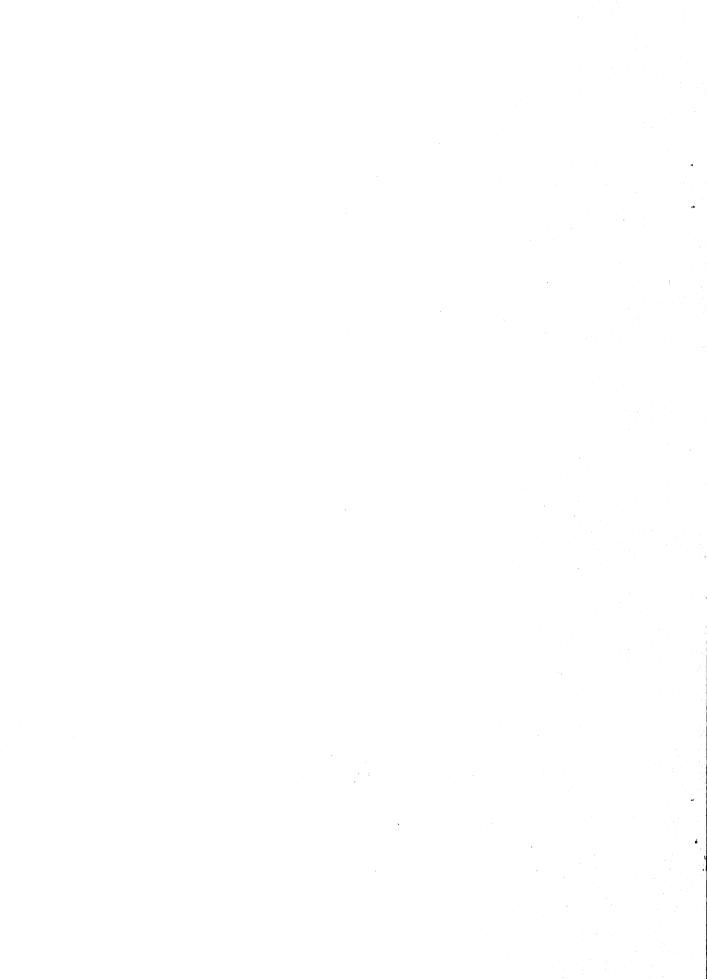
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ABSTRACT

The radiations generated in a nuclear reactor are useful not only in nuclear physics research but also in many nonnuclear fields of science and technology. A review of present and potential applications of the NRL 1-MW reactor is undertaken with the purpose of encouraging additional utilization of this facility.

Neutron activation analysis, a technique capable of assaying very small amounts of many of the chemical elements, is widely used, with examples of its use ranging from the analysis of trace impurities in highly purified materials to law-enforcement purposes. The sensitivities of the neutron activation technique, expressed as the smallest measurable amount of each chemical element, are included.

Other techniques discussed include the measurement of magnetic and crystallographic structure by neutron diffraction, inert and radioactive tracer techniques, the production and use of radioactive sources, radiation effects, and thermal-neutron radiography.

PROBLEM STATUS

This is a final report on one phase of the problem; work on other phases continues.

AUTHORIZATION

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REACTOR UTILIZATION IN SUPPORT OF RESEARCH AND TECHNOLOGY

INTRODUCTION

Experience in scientific research has shown that the problem of communication between different disciplines is a most difficult one and that, consequently, many potential benefits which would result from using techniques and methods of one discipline for research in another are not realized as quickly as desirable. It has frequently been true that the interdisciplinary approach has proven most important, not only for solving a given problem but also for opening entire new areas of research. In nuclear physics research, excellent communication exists with many non-nuclear areas, such that the results and the developments occurring in nuclear research are being applied promptly in those areas. However, there exist many other areas where potentially useful nuclear techniques are neither well known nor being applied to greatest advantage.

In the hope of stimulating a wider exploitation of nuclear techniques, NRL Report 6599 has considered the potentials of the applications made possible by the nuclear facilities at NRL (1). The present report is an extension of one part of NRL Report 6599, in that more detailed considerations are given to applications of a nuclear research reactor in non-nuclear problems. It is, however, somewhat broader, because the use of reactors in nuclear research is briefly discussed.

In dealing with the complex interrelations of the available techniques and their possible applications, a brief description of the more important reactor characteristics and facilities is given, followed by a discussion of the more important techniques made possible by using the reactor. This is followed by a review of several applications in the physical sciences and technology. While many references are given, no comprehensive literature search has been attempted.

NUCLEAR REACTOR FACILITY

The nuclear reactor facility includes a 1-MW thermal research reactor and its auxiliaries. The layout of the facility is shown in Fig. 1, and the floor plan is shown in Fig. 2. The reactor core is suspended below the reactor bridge and is usually operated in the niche position adjacent to the beam ports; it can, however, be operated at any position along the center line of the pool. Vertical shielding is obtained primarily from the 16 ft of water above the core, and horizontal shielding is obtained by the special concrete shield and by the pool (2).*

The light water moderated and cooled reactor core is an intense source of neutrons and gamma rays. The neutrons have a continuous energy spectrum ranging from less than 10^{-3} eV to more than 10 MeV. The neutron energy distribution approximates that of the fission spectrum $(3)^{\dagger}$ at higher energies and in the intermediate energy range, the

^{*}Only those features of the reactor facility deemed important to the following text are described here. A somewhat more comprehensive description of the facility is found in Appendix E of Ref. 1 and in Ref. 2.

[†]The energy spectra of prompt neutrons resulting from fission varies somewhat with the fission process involved. For the thermal fission of U-235, 70% of the neutrons have energies greater than 1 MeV, and 40% have energies greater than 2 MeV. For more detail see Ref. 3.

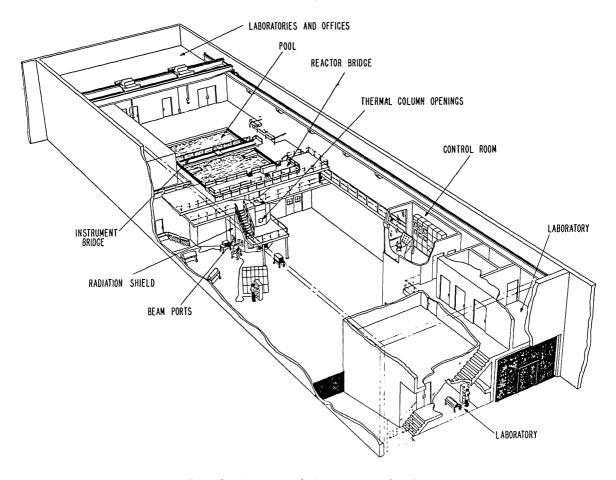


Fig. 1 - Layout of the reactor facility

distribution is characteristic of the slowing down of neutrons in the (light) water and fuel matrix. The most useful component of the flux, the thermal flux, has an energy distribution which is roughly that of a gas at the ambient temperature of the core. For irradiation adjacent to the core, the thermal flux is about 10^{13} n/cm²-sec. The epithermal flux, which includes all neutrons more energetic than the thermal component, is also about 10^{13} n/cm²-sec; about one fourth of this flux has neutron energies greater than 1 MeV.*

Gamma rays are also generated in and near the reactor core. At the licensed power of 1 MW, the dose rate near the core is approximately 4×10^7 rad/hr (roughly a gamma-ray dose rate of 10^{13} MeV/cm²-sec). This dose rate is roughly proportional to the reactor power; however, it has a small delayed component due principally to the radio-active decay of fission fragment nuclei. This intense source of gamma rays can be useful in radiation-damage studies, but frequently it is an undesirable condition associated with neutron exposures.

Neutron Irradiation Facilities

The Pneumatic Rabbit — With the pneumatic rabbit, small sample holders may be transported by air pressure to a region near the reactor core where the thermal neutron

^{*}Accurate measurement of the neutron energy spectrum is difficult, and no serious attempt has been made to determine that of the NRL reactor.

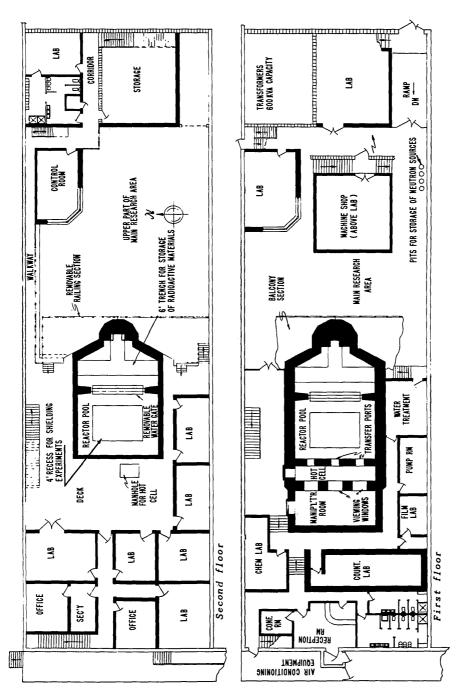


Fig. 2 - Floor plans of the reactor facility

flux is approximately 10^{12} n cm⁻² sec⁻¹. After activation for a few seconds or longer, air pressure delivers the sample holder to any one of four receiving stations within the building. Two stations are located on the balcony just above the thermal column; the third station is in the wall of the Chemistry Laboratory and is accessible from either side of the wall, and the fourth station is on the second floor, adjacent to the laboratories. The sample-holder interior is 13/16 in. in diameter and 3-1/8 in. long.

The High-Flux Exposure Tubes — Several aluminum tubes extend above the surface of the reactor pool from one face of the reactor core. Samples in these tubes near the core are exposed to a thermal-neutron flux of approximately 10^{13} n cm⁻² sec⁻¹ and to a fast-neutron (or epicadmium) flux of approximately the same magnitude. These exposure tubes are normally air filled, but one water-filled tube is available for those experiments in which it is important to keep the temperature near ambient. The inside diameter of the largest tube presently limits the diameter of a sample and its encapsulation to 1.75 in.

The Thermal-Column Exposure Tube — In the center of the reactor shield, on the beam port side of the niche, is a stack of graphite blocks called a thermal column; it is roughly 3-1/2 ft by 4 ft horizontally and 9-1/2 ft high. Graphite, a dense form of carbon, is a good material for slowing down neutrons to thermal energies. An exposure tube 3 in. in diameter, with access available from the balcony (Fig. 1), has been installed into the thermal column. Here samples may be exposed to a neutron flux for which the ratio of thermal neutrons to fast neutrons is much greater than that obtainable near the core. Calibration measurements (4) made as a function of position in the exposure tube established the presence of thermal fluxes up to 10^{10} n cm⁻² sec⁻¹ with a cadmium ratio (gold) exceeding 400.

Beam Port Facilities

The seven horizontal beam ports, shown in Fig. 3, penetrate the concrete and carbon and provide sources of fast neutrons and thermal neutrons. In addition, there is a "through hole" passing alongside the core, which is convenient for certain experiments.

The Hot Cell

A room called a hot cell, shielded by heavy concrete, adjoins the pool at the end opposite the shield. Various metallurgical, chemical, or mechanical operations can be conducted on highly radioactive specimens, up to the kilocurie range, in this room by scientists working with special manipulators from an adjoining room. Objects made radioactive by suspending them, in the pool, close to the reactor core may be transferred directly into the hot cell through a lock-type underwater transfer chamber.

The cell is equipped with three windows for direct viewing. Each window consists of two sheets of 1-in.-thick plate glass with the intervening, 3-ft space filled with a saturated solution of zinc bromide, which is a dense liquid with good optical properties and an excellent shield against radiation. A pair of master-slave hand manipulators is in front of each window, and the cell also has a heavy-duty powered manipulator operated electrically from a console.

Chemistry Laboratory

One laboratory room, equipped with two fume-hoods, is available where chemical operations can be conducted on radioactive materials. One terminal of the pneumatic-rabbit system is available in the wall of this room.

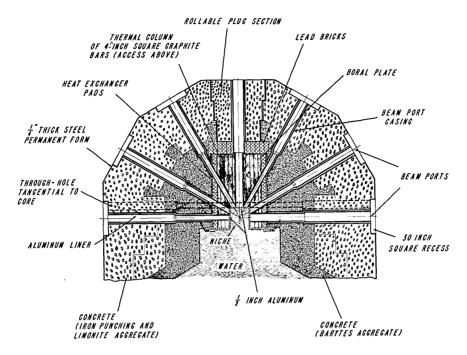


Fig. 3 - Horizontal cross section through the shield, showing the location of the beam ports and the graphite thermal column

TECHNIQUES

Neutron Activation Analysis

Neutron activation analysis consists of the identification and the quantitative measurement of elements present in a sample by inducing characteristic radioactivities by neutron irradiation. The sensitivity of this method is such that it is most commonly used for measuring elements present only in trace quantities; however, it has been used in accurate quantitative analyses of the more important constituents of alloys. Trace elements may be present in a sample as extraneous impurities, or they may be purposely introduced as a stable isotope tracer. This last technique is similar to the radioactive-tracer technique; it can, however, be used in many cases where radioactive tracers are not desirable or permissible. Frequently, nondestructive analyses are possible; however, concomitant chemical separations are sometimes helpful or necessary.

The sensitivity of the analytical method varies radically from element to element; with some elements less than 10^{-12} g are detectable, while with others the method is valueless. The situation is sometimes complicated by interference from other elements in the sample, and several choices of the instrumentation used to measure the radioactivity created are available. Consequently, most analyses become special cases requiring individual investigation.

One important question is how the sensitivity of this technique compares with alternate techniques. Dr. V. P. Guinn, an advocate of neutron activation analysis, states that, 'for more than half of the elements of the periodic system, high flux neutron activation analysis is the most sensitive method known' (5).*

^{*}It is implied that the "high flux activation" involves irradiation in a thermal flux of 10^{13} n/cm^2 -sec.

Neutron activation analysis is widely used, and at least two laboratories are performing analyses on a commercial basis (6).*

Neutron Reactions and Measurements Methods — The most frequently used nuclear reaction is thermal-neutron capture (the (n,γ) reaction) which generally leads to neutron-rich isotopes. These may be stable (and thus valueless), or they may decay by emitting a beta particle or one or more gamma rays, or both. The (n,p), (n,α) , and (n,2n) reactions are also used; these usually occur only when fast neutrons are incident on the target material. In a few instances, secondary reactions are employed, an example being the $^6\text{Li}(n,\alpha)$ t reaction followed by the $^{16}\text{O}(t,n)^{18}\text{F}$ reaction. It is then necessary to have lithium and oxygen in contact, and the amount of ^{18}F formed will depend on the concentrations of both lithium and oxygen.

The resultant radioactivity can be assayed by measuring gamma rays, beta particles, or characteristic x rays. The most widely used technique is gamma-ray measurement. The principal advantages are that self-absorption in the sample is generally not troublesome, and the discrete gamma-ray energies can be used to assay, simultaneously, several different radioactivities present in a given sample. Sometimes, however, the radioactive isotope of interest emits no gamma rays, or relatively few, making this approach useless or of low sensitivity. The use of beta particles suffers from self-absorption in the sample and from severe interference if two or more beta activities are present simultaneously; however, beta detection sensitivity is high. The measurement of characteristic x rays is not widely used. It is difficult, if not impossible, to use it for low-Z elements, and self-absorption will usually be much more severe than with gamma rays; however, it is sometimes preferable (7).

The instrumental method of neutron activation analysis is presently very popular. It consists of the nondestructive analysis of a sample by irradiation and subsequent assay by gamma-ray measurements. It is effective for a wide range of samples, and it is comparatively rapid and convenient. Its usefulness has been greatly enhanced by improvements in gamma-ray spectroscopy, including computer stripping of pulse-height spectra. The NaI(T1) scintillation spectrometer has been generally used in this work; however, the higher resolution, germanium-lithium-drifted (Ge(Li)) detector will be more widely used as larger detectors become more available.

Interferring Radioactivities - Two kinds of interference from other elements in a sample can occur. The first is caused by the formation of the radioactivity of interest by a different nuclear reaction, and the second is caused by different radioactive species. An example of the first type of interference may be seen when sodium is to be analyzed in a matrix containing aluminum. If the irradiation is carried out near the core, the thermal flux leads to the desired reaction, 23 Na(n, γ) 24 Na (t $_{1/2}$ = 15 hr). Concurrently, fast neutrons with energies greater than the threshold value of 3.26 MeV lead to the reaction 27 Al(n, α) 24 Na. While the second reaction is less probable than the first, if the sodium content is low, its contribution to the ²⁴Na activity can be obscured by the activity from the aluminum. If the interference is not great, analysis may be possible by comparing two samples, one irradiated bare and the other cadmium covered. The bare sample gives the combined contribution of both reactions, and the cadmium-covered sample gives, essentially, the aluminum contribution. An alternative approach is to use irradiations in the thermal column, where the ratio of thermal neutrons to fast neutrons is greatly enhanced. The thermal flux is about three orders of magnitude lower in the thermal column than it is near the core, and the detection sensitivity suffers accordingly.

^{*}Among those offering such analytic services are Union Carbide Corp., Nuclear Division, Tuxedo, N. Y., and General Dynamics, General Atomic Division, San Diego, Calif. The latter offers a wire service in which pulse-height spectra (400 channels) are printed out in the customer's laboratory.

When considering the analysis of a given element, refer to Koch's handbook (8), which lists the interfering reactions that may be expected. Estimates of the severity of interference can usually be made using estimates of the fast-neutron cross sections and the computational techniques given by Roy and Hawton (9). Alternatively, activations with known amounts of the desired and the interfering elements may be made, and the severity of the interference accurately determined.

The second kind of interference depends both on the intensities of the radiations from the desired and undesired radioactivities formed from elements in the sample and the ability of the detector used to discriminate between the radiations involved. In gamma-ray measurements, the newer Ge(Li) solid-state detectors are far superior to the older NaI(Tl) scintillation detectors, regarding the ability to separate gamma rays of different energies, as shown by Fig. 4 (reprinted from Ref. 1, p. 22). The addition of an anti-Compton mantle around a Ge(Li) detector would also lead to additional improvement. For situations where a single gamma-ray detector will not suffice, coincident gamma-ray measurements can be helpful if the desired radioactive isotope emits a positron or, alternatively, two or more gamma rays in a prompt cascade. Another approach which may be helpful is to perform a chemical separation on the irradiated sample, when this separates the desired radioactive nuclides from the interfering ones.

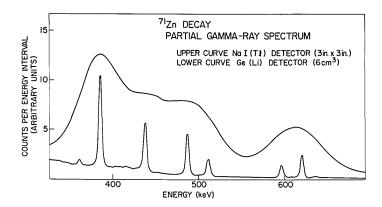


Fig. 4 - A comparison between the pulse-height spectrum obtained with a sodium iodide scintillation detector and that obtained with a lithium-drifted germanium detector

Sensitivity — Sensitivities are best quoted as the smallest amount of an element which can be detected, rather than (as customary) the relative concentration (such as ppm or ppb), remembering that there will be some limit, usually not restrictive, to the maximum size of the sample which can be employed. This limit is determined by self-shielding, within the sample, of either the neutrons during irradiation or the radiation emitted during the measurement of the radioactivity. In some cases the limit is determined by the size of the sample available.

Table 1 summarizes the sensitivities (the smallest amounts detectable) of various elements. Koch (8) has computed "standard sensitivities" for certain conditions; these have been adjusted to conditions obtainable with the NRL reactor irradiations, followed by gamma-ray detection, to obtain the entries in column 3. In column 3 are the masses, in micrograms, of each element which, when irradiated to saturation or for a maximum of 30 days, will have a radioactivity created by the (n, γ) reaction, which emits 1000 gamma rays per minute. Column 4 shows the sensitivities quoted by the Air Force

Table 1 Neutron Activation Analysis Sensitivities

]			Quoted	d Sensitivities	(μg)*
Atomic Number	Element	NRL	NETF		Other
Z		NKL	NEIF	Sensitivity	Method†
1 2	H He		_	-	
3	Li		_	3×10^{-4}	⁶ Li(n, α) ³ H; β ^{-‡}
3 4	Be			4×10^{-4}	$^{9}\mathrm{Be}(\mathrm{n},\alpha)^{6}\mathrm{He};\beta^{-}$
5	B		_	0.15	$^{11}B(n,\alpha)^{8}Li;\beta^{-}$
5 6	Ĉ				
7 8	N		_	1.3	$^{14}N(n,2n)^{13}N; \beta^{-}$
8	0	-	-		. -
9	F	_	_	6×10^{-3}	19 F(n, α) 16 N; β
			_	9×10^{-3}	19 F(n, α) 16 N; γ
10	Ne		– , i	0.6	20 Ne(n,p) 20 F; β^- or γ
11	Na	6×10^{-4}	3.5×10^{-4}		
12	Mg	2×10^{-2}	3×10^{-2}		
13	Al	4×10^{-4}	5×10^{-5}	-	
14	Si	3	5×10^{-2}	2×10^{-2}	β-
15	P		1×10^{-3}	3×10^{-3}	β- 31.D(
10	a		-	$\begin{array}{c c} 6\times10^{-3} \\ 0.2 \end{array}$	β^{31} P(n,p) ³¹ Si; β^{+} or γ
16 17	S Cl	$\begin{array}{c c} 4 \\ 1.5 \times 10^{-3} \end{array}$	$0.2 \\ 1.5 \times 10^{-3}$	7×10^{-4}	β - β -
18	A	2×10^{-4}	1.5 × 10 -	7 × 10	P
19	K	3×10^{-2}	4×10^{-3}	6 × 10 - 3	β-
20	Ca	7×10^{-2}	0.19	0 × 10	
21	Sc	3×10^{-5}	1×10^{-4}	1×10^{-4}	β-
22	Ti	2×10^{-2}	_		
23	V	2×10^{-3}	5×10^{-5}	3×10^{-5}	β-
24	${f Cr}$	3×10^{-2}	1×10^{-2}	2×10^{-3}	β-
25	Mn	1 × 10 - 5	3×10^{-5}		
26	\mathbf{Fe}	0.5	0.45	_ ,	_
27	Co	1×10^{-5}	1×10^{-3}	2×10^{-3}	β-
28	Ni	2×10^{-2}	1.5×10^{-3}	1×10^{-3}	β-
29	Cu	1×10^{-3}	3.5×10^{-4}	4 × 10 - 4	x rays
30	Zn	3×10^{-2}	2×10^{-3}	1×10^{-3}	β-
31	Ga	3 × 10 -4	3.5×10^{-4}		
32	Ge	2×10^{-3}	2×10^{-3}	1×10^{-4}	x rays
33	As	3 × 10 - 4	1×10^{-4}	1.5×10^{-4}	β-
34	Se	1×10^{-2}	2.5×10^{-3} 1.5×10^{-4}	9×10^{-4} 5×10^{-5}	β- 1 8+ and 8-
35 36	Br Kr	1.5×10^{-4} 5×10^{-5}	1.0 710	$\begin{array}{c c} 5 \times 10 \\ 7 \times 10^{-3} \end{array}$	β^+ and β^-
37	Rb	3×10^{-2}	1.5×10^{-3}	7×10^{-3}	β
38	Sr	2×10^{-3}	3×10^{-2}	0.9	β^{-}
39	Y	4	5×10^{-4}	9 × 10 - 4	β-
40	\mathbf{Zr}	3 × 10 - 2	1.5×10^{-2}		
41	Nb	3 × 10 -4	0.5	1.5 × 10 - 2	β-
42	Mo	1 × 10 - 2	5×10^{-3}	_	l ·
43	Tc	No stable	isotope		_
44	Ru	3 × 10 - 3	5×10^{-3}	_	-

(Table continues)

Table 1 (Continued)

			Quoted	l Sensitivities	(μg)*
Atomic Number	Element	MDY	MEMBE		Other
Z		NRL	NETF	Sensitivity	Method†
45	Rh	2×10^{-5}		_	
46	Pd	3×10^{-4}	2.5×10^{-4}		
47	Ag	3×10^{-4}	5.5×10^{-3}	1 × 10-5	β-
48	Cd	4×10^{-3}	2.5×10^{-3}		·
49	In	2×10^{-6}	5×10^{-6}		
50	Sn	3×10^{-2}	1×10^{-2}	-	
51	Sb	6×10^{-4}	2×10^{-4}	4×10^{-4}	β^{-}
52	${f Te}$	5×10^{-3}	5×10^{-3}		·
53	I	4×10^{-4}	1×10^{-4}	6×10^{-5}	β-
54	Xe	3×10^{-2}	_		·
55	Cs	2×10^{-3}	1.5×10^{-3}		
56	Ba	4×10^{-3}	2.5×10^{-3}	1×10^{-3}	 β ⁻
57	La	2×10^{-4}	1×10^{-4}	· _	`
58	Ce	2×10^{-2}	5×10^{-3}	- .	
59	Pr	4×10^{-3}	1×10^{-4}	1 × 10 ⁻⁴	β-
60	Nd	2×10^{-3}	5×10^{-3}	_	
61	Pm	No stable	e isotope	_	
62	Sm	5×10^{-5}	3×10^{-5}		<u></u> -
63	Eu	6 × 10 ^{-7§}	1.5×10^{-6}	2×10^{-7}	β-
64	Gd	2×10^{-3}	1×10^{-3}	1×10^{-3}	β-
65	Tb	2×10^{-4}	2×10^{-4}	_	
66	Dy	3×10^{-6}	1.5×10^{-6}		
67	Но	6 × 10 - 5	2×10^{-5}	3×10^{-5}	β-
68	Er	6×10^{-4}	1×10^{-3}		
69	Tm	4×10^{-4}	1×10^{-4}	9×10^{-5}	β-
70	Yb	3×10^{-4}	1×10^{-4}	9×10^{-5}	β-
71	Lu	2×10^{-5}	1.5×10^{-5}	2×10^{-6}	β-
72	Hf	2×10^{-4}	1×10^{-3}		
73	Та	1×10^{-3}	3.5×10^{-4}	-	
74	W	3×10^{-4}	1.5×10^{-4}	2×10^{-4}	β-
75	Re	2×10^{-4}	3×10^{-5}	4 × 10 ⁻⁵	β-
76	Os	1×10^{-3}	1×10^{-3}		Į.
77	Ir	5 × 10 - 6	1.5 ×10 ⁻⁵	2×10^{-5}	β^{-}
78	Pt	5×10^{-3}	5 × 10 ⁻³	2×10^{-3}	β-
79	Au	2 × 10 - 5	1.5×10^{-4}		
80	Hg	8 × 10 ⁻⁴	6.5×10^{-3}	4 ×10 ⁻³	β-
81	Tl	2 ¶	3 ×10 ⁻²	8×10^{-3}	β-
82	Pb	_	1 ×10 ⁻¹	2.5	β-
83	Bi		2 ×10 ⁻²	0.14	β-
90	Th	3×10^{-4}	_	9 ×10 ⁻⁵	β-
92	U	2 × 10 - 4	5×10^{-4}	_	_

^{*}See text for the conditions assumed.

[†]The code is as follows: (a) β^- , β^+ , x rays; activation by the (n, γ) reaction and assay by measurement of beta particles or x rays (b) for methods of the type ${}^6\text{Li}(n, a){}^3\text{H}$; β^- , activation by the indicated nuclear reaction ${}^6\text{Li}(n, a){}^3\text{H}$ and assay by measurement of the radiation indicated by the symbol β^- .

The β from the decay of ³H has a very low energy (18 keV maximum); consequently, assay is difficult.

[§] The value given is for assay with x rays. The energy of the $k\alpha$ line is approximately 41 keV. The value given is for assay by x rays. The energy of the $k\alpha$ line is approximately 71 keV.

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Nuclear Engineering Facility Staff (10). These apply to a thermal-neutron exposure rate of 10^{13} n/cm²-sec and irradiation to saturation or for 30 days, whichever is shorter. No information is given about the method of assay to be used or the initial disintegration rate assumed. The conditions assumed in deriving the sensitivities shown in columns 3 and 4 are not identical; thus, some differences in sensitivities are to be expected.

Although not usually shown, the minimum detectable masses which apply when beta counting is employed are equal to or smaller than those for gamma-ray detection; when the beta sensitivity differs markedly from the gamma sensitivity, it is given in column 5.

For some elements not readily measured by activities produced by thermal neutrons, sensitivities expected by other approaches, and the technique involved, are shown in columns 5 and 6.

Neutron Beams

The neutron flux in the reactor core is essentially isotropic in direction and has the very broad energy spectrum characteristic of a light-water-moderated reactor. Beams of neutrons can be formed by collimators directed toward the core or some scattering material near the core. Furthermore, it is possible to select neutrons of a given small energy increment by diffraction from a suitable crystal. The flux is reduced by these processes, and the reduction depends principally upon the spectral intensity of the neutrons from the reactor at the energy selected and the width of the energy increment. In addition, it is possible to polarize the neutrons in a beam, when this is advantageous. These techniques permit a wide spectrum of experiments, a few of which will now be discussed.

Neutron Diffraction Measurements — Thermal neutrons have wavelengths generally comparable to the interatomic distances between atoms in a solid, and neutron diffraction measurements analogous to x-ray diffraction measurements are widely employed. Generally, the neutron technique does not replace the x-ray technique for crystallographic studies; it is mainly used in situations where neutron diffraction will furnish additional information. The interaction of neutrons with atoms differs from that of x rays in several important respects. First, the x-ray cross section increases monotonically and fairly rapidly with the atomic number Z of the diffracting atom, while the neutron cross section is roughly independent of Z; therefore, neutron diffraction is widely used in studies of crystals containing both high-Z and low-Z elements. This ability has been exploited in a study of the anomalous behavior of both palladium hydride and palladium deuteride which occurs near 50°K. It was found that one hydrogen atom in each unit cell moves to a new position at this temperature, causing a change in some of the characteristics of the material (11).

The neutron possesses a magnetic moment, and, consequently, the neutron-diffraction technique is capable of probing the magnetic structure of solids. This technique has been used at NRL to study the magnetic structure of several crystals (12-14) and in the pioneering study of spin waves (15-17).

The third difference between neutron diffraction and x-ray diffraction is that neutrons usually penetrate more deeply than x rays into the sample under study, thus giving information more characteristic of the bulk property of the sample. This characteristic of neutrons is being exploited in a program of neutron-diffraction measurements of glasses and liquids.

Measurement of Spin States of Neutron Resonances — In a pioneering experiment conducted at NRL, the spin of the compound nucleus formed by resonant neutron capture has been measured for a considerable number of neutron resonances in several target

nuclei (18,19). By polarizing both the nuclear sample and the neutron beam, one can obtain the spin of the compound nuclear state by observing the change in transmission of the beam when the neutron polarization direction is reversed.

Neutron Capture Studies — The gamma rays emitted following radiative capture of neutrons have been studied by Vogt (20), who used a monochromatic neutron beam and, more recently, by Ritter and Namenson, * who used unanalyzed neutrons. In addition, the known gamma-ray spectrum of yttrium has been used as a calibration source for Ge(Li) detectors.

Production of Radioactive Sources

Radioactive sources can be created by irradiations in the reactor. The purchase of sources from a commercial supplier may be preferable in some cases, particularly when the sources are obtained by chemical separation from reactor waste material and/or when high-specific-activity sources are required. Alternatively, the production of sources in the NRL reactor is particularly appropriate for short-lived activities, when travel time from the production source to the user is crucial. Usually, radioactive elements produced in the reactor will be an isotope of the target material, and the specific activity obtainable will be set by the reactor flux and reasonable irradiation times.† It is easy, however, to produce a wide variety of sources useful for instrument calibration and for many radioactive tracer applications.

The energy levels and decay schemes of radioactive nuclei produced in the reactor are determined by nuclear spectroscopy methods as a part of the research program of the Reactors Branch. The use of the rather newly developed solid-state detectors makes it possible to determine energy levels and decay schemes with a completeness and precision previously impossible. The more valid data now becoming available are useful both in applied work, such as neutron activation analysis, and in the detailed testing of theories of nuclear structure. The recent study of ⁸³Se (21,22) demonstrates both the complexity of the decay process of this isotope and the ability of the newer instruments.

Tracer Analysis

Two nuclear techniques for tracing chemical or physical processes are (a) neutron activation analysis, in which an appropriate inert trace element is employed during the process under study and (b) use of a radioactive substance as a tracer. Use of an inert tracer has the advantage (often of crucial importance) that no nuclear radiation is present during the process under study; however, it must be possible to collect samples of the inert tracer for subsequent analysis. One advantage of the radioactive-tracer approach is that the tracer is different from the inert element; consequently, the self-diffusion of a single-isotope element can be measured by a radioactive tracer, although no inert tracer would be available.

Radiation Effects

The various radiations available in the reactor can be used in radiation-effects studies. Samples placed near the core are simultaneously subjected to slow and fast neutrons

^{*}Work now in progress by J. Ritter and A. I. Nameson of the Linac Branch, Nuclear Physics Division.

[†]If the radioactive species desired can be formed by an (n,p) or a (n,α) reaction, chemical separation from the inert target material can be performed generally leading to a higher specific activity source. It will usually be necessary for the experimenter to perform the chemical separation.

and to gamma rays. In some radiation-effects studies, two or more processes may occur simultaneously during such irradiations, making interpretation difficult; however, in others, the mixed fluxes may be satisfactory or even desirable.

The susceptibility of materials and devices to radiation varies widely; therefore, the total dose available in reasonable times at the NRL reactor is adequate for many purposes. Various techniques make it possible to control the irradiation conditions. The thermal flux incident on a sample can be drastically reduced by placing the sample inside a thin-walled cadmium enclosure. Selective shielding can somewhat enhance fast neutrons relative to gamma rays, or vice versa. Liquid or gas samples containing both the material to be irradiated and a fissile element may also be irradiated; in this case the kinetic energy of the fission fragments is delivered directly to the material in which it is immersed.

Among the interesting radiation effects are the synthesis of chemical compounds, changes of the properties of materials, and both transient and permanent changes in the operating characteristics of devices and equipment.

APPLICATIONS

Some of the more important applications of a research reactor will now be outlined. While the majority of the applications discussed have been or are being conducted at NRL, others are included for completeness. No attempt has been made to assess their relative importance or to list all possible applications.

Studies of the Atmosphere

Study of Air Movements — A technique which permits the measurement of atmospheric motions has been developed at NRL (23). A stream of aerosols containing indium is dispersed from an airplane, and on subsequent transits, the aerosols are collected on a moving filter. The aerosols collected at a given spot on the filter can be related to time and to the position of the plane when the sample was collected. The fraction of the dispersed aerosol collected at a given location is then determined by neutron activation analysis. Laboratory and field measurements established the feasibility of the method; a major problem was directing the airplane to a known position relative to the track of the aerosol initially dispersed.

Similar techniques have been used by others to determine the distribution on the ground of effluents from industrial stacks (24); a study of the distribution in the air during transit also appears feasible.

Distribution of Natural Trace Elements in the Atmosphere — Neutron activation analysis measurements of naturally occurring atmospheric aerosols collected both at ground level and from an airplane have been made at NRL (25). Also, when the Surtsey volcano erupted, several aerosol samples were collected in and near the cloud above the volcano and were analyzed (26). One surprising result was the high concentration of manganese in all the samples; this concentration in the ambient atmosphere was many orders of magnitude higher than expected if it were primarily of marine origin.

Another interesting phenomenon found in one set of measurements was the strong dependent of aerosol concentration on altitude. Thus, it would appear that a study of naturally occurring aerosols may improve our understanding of meteorological phenomena, in particular the inversion conditions contributing to the severity of air pollution. Other investigators (27) have shown that the analysis of Cl, I, and Br in natural aerosols can lead to information on the atmospheric concentration of the leaded additives of automotive gasoline, a matter of concern in air pollution.

Oceanographic Studies

Naturally Occurring Trace Elements — Neutron activation analysis of trace elements in the oceans is a fruitful field of endeavor, and several programs are underway.* Knowledge of the variation of concentration in the water should be useful in understanding the physical, chemical, and biological processes which occur. Perhaps of greatest interest are phenomena occurring at and near the surface and the bottom of the ocean. It is known, for example, that the ratios of the concentrations of Cl, I, and Br in marinegenerated airborne aerosols are drastically different from those in the ocean (27), and the mechanisms involved are not understood.

Composition of the Ancient Sea — It has been postulated that the composition of the sea in past eras may be deduced by first establishing a relation between the concentrations of several elements in barnacles and the chemical composition of the water in which they grew and, subsequently, measuring the concentration of the same elements in barnacles from old deposits of known age. A program is in effect at NRL in which the concentration of Na, Ca, Si, Mg, and Mn in appropriate samples is measured by neutron activation analysis.†

Tests of Highly Purified Materials

In efforts to develop highly purified materials, several analytical techniques are useful, since no single technique is likely to have adequate sensitivity for all the impurities of interest. Thus, it is not surprising that neutron activation analysis is one of the tools employed. To cite a few examples, the National Bureau of Standards has analyzed, with the NRL reactor, several 'pure' metal samples, both as standards in their analytical studies and as a part of their efforts to produce successively purer samples (28). Also, IBM (29)‡ and Texas Instruments, Inc. (30) use neutron activation analysis to determine trace impurities which degrade the performance of solid-state devices.

Analysis of Standard Reference Materials

The National Bureau of Standards is investigating the use of neutron activation analysis as a possible method of determining the elemental composition of several Standard Reference alloys. In some cases studied, the activation-analysis results, using the NRL reactor, appear to be more precise than those obtained by wet-chemistry techniques. In determining the amount of selenium in the reference material 1170 selenium steel, the neutron activation result was $0.293\% \pm 0.003$ (the ± 0.003 value is the standard deviation of the individual values). In comparison, wet-chemistry measurements by three independent groups were 0.298%, 0.288%, and 0.290% (28).

Diffusion in Solids

Radioactive tracers have been widely used in studying the diffusion of elements into solids (31). This approach has one unique advantage; namely, the radioactive tracers can be distinguished from other atoms of the same element. Thus, self-diffusion can be

^{*}Programs in this area are being conducted by Texas A&M University, by the Marine Science Department of the Naval Oceanographic Office, and by the Department of Geology of Yale University.

[†]Work is now underway in the Nuclear Oceanography Branch, Ocean Science Division. The chief investigator is C. M. Gordon.

IBM uses both neutron activation analysis and radioactive tracer techniques. A summary of their laboratory and its work is given in Ref. 29.

studied even in an element with a single isotope, a procedure obviously impossible with inert tracers.

In circumstances where inert tracers can be used (e.g., when one isotope is diffused into a matrix not containing that isotope), neutron activation analysis can be useful if serial sectioning of the sample after diffusion and prior to analysis is permissible. In some cases it may be possible to use accelerator techniques to determine, with high sensitivity, the concentration versus depth in the sample in situ.*

If it is desired to examine the composition of the fine-grain structure in a sample, the above techniques are not expected to be adequate; in such cases the microprobe approach would appear to be ideal.†

Study of Corrosion

Boiler corrosion in a conventional steam plant is inhibited by the formation of a protective film composed principally of iron and oxygen. The addition of dissolved alkali metals to the boiler water plays an important role in the effectiveness of these protective films. In a program now underway at NRL, the amounts of Li, Na, and K present in protective films formed under different operating conditions are being measured by neutron activation analysis. In determining the amount of Li present, the secondary reaction discussed above is used, in which tritons from a lithium-neutron reaction form F-18 by a secondary reaction with oxygen.

Radiation-Induced Chemical Synthesis

In some instances synthesizing chemical compounds by nuclear radiation may be more suitable than by other methods. It is often desirable to have a rapid quenching mechanism operative, to inhibit the reverse reaction. Such quenching is expected when the reactants are subjected to nuclear irradiation, since energy is delivered largely to individual molecules, which may react in the desired fashion, while the surrounding medium is at ambient temperature and furnishes rapid quenching.

In an attempt to form a nitrogen-fluorine compound which would be useful as a rocket-fuel oxidizer, a nitrogen-fluorine mixture was exposed to the NRL reactor flux. Small quantities of NF $_3$ were synthesized; however, the yield was not high enough to make such a synthesis practicable (32).

In a comprehensive review article (33), \S Meyer Steinberg has summarized the major research programs in which nuclear energy is considered for use in chemical synthesis. Research aimed toward future applications to the fixation of nitrogen and the production of synthesis gas (CO + H₂), hydrogen peroxide, and hydrazine is reviewed, and the more advanced radiation processing of polymers is discussed.

Miscellaneous Tracer Applications

No attempt will be made to summarize the widespread use of both inert and radioactive tracers. A few suggested applications, not covered elsewhere, are outlined to indicate the range of potential uses in the physical sciences and in technology.

^{*}See Ref. 1, Section III, 3b, page 9.

[†]The X-Ray Optics Branch, Nuclear Physics Division of NRL played a very important role in the development of the microprobe instrument and methods of microprobe analysis. †This study is apart of the research program of the Physical Chemistry Branch, Chemistry Division. The chief investigator is T. A. Kovacina. \$Includes extensive references.

Flow Measurements — Tracers have been used to measure the flow rates (34)* in industrial plants, pipelines, and rivers and to measure ocean and river currents at a given point (35). \dagger

Filter Systems — Tracers have been used extensively to test the efficacy of filter systems. Aerosol tracers detected by a light scattering cell are usually used in testing particulate filters (36-38). Activated charcoal filters, used to absorb gaseous effluents, have been tested by releasing either radioactive or inert iodine into the exhaust stream while samples are collected on smaller charcoal filters from the air stream before and after it enters the filter (39,40). Radioactive-iodine sources produced by the NRL reactor have been used by the National Bureau of Standards in tests of their filter systems. Recently, an inert-iodine tracer has been used to test the NRL reactor filter system.

Quality and Process Control

The Naval Ordnance Station is investigating the feasibility of using neutron activation analysis with the NRL reactor in the quality control of rocket-propellant manufacture. It has been found that this method is more accurate than other methods.

The widespread use of nuclear techniques includes the use of beta-ray and gamma-ray gages to measure the thickness and/or the density of sheet material, cigarettes, etc. Radioactive tracers have been used to determine how much residual solder flux remains on automatically-processed electronic circuit boards (41), an important consideration in ensuring against failure from corrosion. Similarly, both neutron activation (29,30) and tracer (29) techniques are used in developing and checking processes used in the fabrication of semiconductor devices.

Calibration Sources

Radioactive sources useful in instrument calibration can be produced by reactor irradiations or obtained commercially, as discussed above. In addition, the neutron fluxes available may be used to calibrate instruments; specifically, the thermal column has been used in calibrating thermal-neutron dosimeters. High-energy gamma-ray sources resulting from the capture of thermal neutrons in yttrium and from the short-lived N-16 activity induced in circulating reactor-pool water have been used to calibrate Ge(Li) detectors for the research program of the Linac Branch. The yttrium source is especially useful, since its spectrum contains about 70 gamma-ray lines covering the energy range up to 8.5 MeV; normally available radioactive calibration sources have gamma-ray energies less than 2.6 MeV.

Law Enforcement and Related Uses

The use of neutron activation analysis for law enforcement has increased rapidly in recent years. Generally, the purpose is to determine whether two or more samples of evidence have the same or different origins; hence, quantitative determination of elemental composition is not usually required, and the ability to compare samples nondestructively is highly advantageous. Extensive forensic programs employing the NRL reactor are conducted by the Internal Revenue Service, the Federal Bureau of Investigation, and the Post Office Department. One example of such use is in determining the origin of narcotics. This can be deduced by the amounts of various trace elements

^{*}This article is an edited reprint of one in the Journal of the British Nuclear Energy Society 3:252 (1964).

[†]This article is a review and contains extensive references.

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remaining in the drugs (42,43). The more common applications are the determination of whether two or more samples such as soil, paint, moonshine, and lubricating oil have the same or different origins (44).

In a related area, trace elements found in representative diets from several areas of the U.S. are measured by the Food and Drug Administration as a part of their Total Diet Monitoring Program. It is hoped to relate diet to questions of health; another important consideration is the possibility of contamination by noxious or poisonous chemicals used as insecticides or weed-control agents.

It is possible to label a product by adding traces of some combination of rare elements which can be easily measured by neutron activation analysis. Such a procedure would be useful in distinguishing labeled products from counterfeit ones. Since about 15 suitable elements are available, an extremely large number of distinct codes are possible. The Internal Revenue Service is considering the use of such coding to label legally produced narcotics, and other uses of the technique are being considered by various industries.

Radiation Effects

The NRL reactor flux has been employed to determine what limitations may be imposed on the use of strain gages in a reactor environment.* In this study, the combined neutron and gamma-ray fluxes were desirable, since they approximated the environment of interest. A study on the effects produced by reactor irradiations on GaAs is being planned.

Thermal-Neutron Radiography

Thermal-neutron radiography is still in the developmental stage, and, generally speaking, it is a more difficult technique than the x-ray method. It has potential value as a technique complementary to x-ray and gamma-ray radiographic methods, since its response to various elements is markedly different. For example, using this method it is possible to detect a stream of water flowing on the opposite side of a lead wall. Various imaging methods are available which have reasonably good spatial resolutions (45,46).

One military application which appears feasible is the nondestructive testing of small explosive charges contained in steel, brass, or lead jackets. It has been found that thermal-neutron radiography will show the condition of the explosive charges in Polaris Missile EBW Detonators and of lead-encased explosive strands (47). This technique has also proved effective for examining clad reactor fuel specimens; radiographs have shown fuel cracking, swelling, and relocation in irradiated test samples (48).

CONCLUSION

The NRL nuclear reactor has been employed extensively to support a wide variety of research, development, and technology programs, which run the gamut from basic research to important direct military and civilian applications. The successful use of the reactor illustrates the usefulness of applying nuclear facilities and techniques to non-nuclear research and to nuclear research and technology. More extensive use is expected to be advantageous to many components of the Laboratory, and a considerable increase is feasible.

^{*}The chief investigator is N. J. Rendler, Ocean Structures Branch, Ocean Technology Division.

Neutron activation analysis is widely used to determine the amount of elements of interest in various samples. The sensitivity of this method varies widely; for some elements it has no value, while, for others, less than 10^{-12} grams can be measured. For roughly half of the chemical elements, this analytical technique is the most sensitive one available, and its use continues to increase.

Other techniques which can be exploited include the use of radioactive sources for instrument calibration, process control, and tracer studies. (Neutron activation analysis can also be employed in inert tracer studies.) Neutron diffraction measurements and radiation effects, including chemical synthesis and thermal-neutron radiography studies, are possible.

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taken with the purpose of encouraging additional utilization of this facility.

Neutron activation analysis, a technique capable of assaying very small amounts of many of the chemical elements, is widely used, with examples of its use ranging from the analysis of trace impurities in highly purified materials to lawenforcement purposes. The sensitivities of the neutron activation technique, expressed as the smallest measurable amount of each chemical element, are included.

Other techniques discussed include the measurement of magnetic and crystallographic structure by neutron diffraction, inert and radioactive tracer techniques, the production and use of radioactive sources, radiation effects, and thermal-neutron radiography.

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